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IMPROVED CERAMIC-BONDED CALCIUM FLUORIDE COATINGS FOR LUBRICATING NICKEL-BASE ALLOYS IN AIR AT TEMPERATURES FROM 500° TO 1700° F

by Harold E. Sliney

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Cleveland, Ohio

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Page 4, item (6), line 7: The sentence beginning "Underfiring is indicated" should read "Underfiring is indicated if the coating has a rough, porous texture; overfiring is indicated if the CaF_2 agglomerates."

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NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

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SUMMARY

This study was conducted with the objective of developing modifications in coating procedure and composition that would improve the lubricating characteristics of ceramic-bonded calcium fluoride (CaF_2) coatings.

The lubricating properties of these coatings were strongly influenced by the details of the coating application procedure. Of particular importance were: thorough wet-grinding (pebble milling) of CaF_2 and the ceramic binder, the use of a very fine spray when applying the milled material to the substrate, and careful control of firing time and temperature when fusion-bonding the coating to the substrate. A 3 percent addition of molybdic oxide (MoO_3) to the coating composition and the application of a rubbed film of a fluoride eutectic composition (62 percent barium fluoride - 38 percent calcium fluoride) were beneficial. Improved performance at 1000° F was also obtained by initially running the specimen at 1500° F to establish a glazed wear track.

Common factors in all beneficial modification were (1) a reduction in the porosity of the coating and (2) an improvement in the homogeneity of CaF_2 dispersion throughout the ceramic binder.

INTRODUCTION

One of the problem areas associated with high Mach number aircraft and reentry vehicles is the effective lubrication of mechanisms such as control surface bearings exposed to a high-temperature oxidizing environment. In air, lubricating oils are temperature limited by oxidative instability (degradation caused by chemical reaction with oxygen). High-temperature oils of current interest are not oxidatively stable above approximately 500° F. The temperature limitations of lubricating oils therefore dictate other types of lubricants above 500° F.

One feasible method of lubricating at higher temperatures is the use of bonded solid lubricant coatings. Previous research at the Lewis Research Center demonstrated the feasibility of using a ceramic-bonded calcium fluoride (CaF_2) coating as a high-temperature solid lubricant for certain nickel-base superalloys, such as Inconel X and René 41. This coating effectively lubricated Inconel X from 500° to 1500° F and René 41 from 500° to 1900° F (refs. 1

and 2). Accumulated experience with this coating, however, showed that from about 500° to 1000° F, the friction coefficients were sometimes objectively high (>0.20) and erratic during the initial phase of the friction experiments. Although in most cases low rider wear was observed, the requirement of a long run-in period with high and erratic friction values could make the CaF_2 coating appear unattractive for some applications where it would otherwise provide effective lubrication.

The objective of the present study therefore was to determine the sources of the erratic run-in characteristics and to determine what corrective measures were in order.

The friction coefficients and the wear-inhibiting properties of the various coating modifications were determined in an air atmosphere at temperatures up to 1700° F. The specimen configuration consisted of a $2\frac{1}{2}$-inch-diameter rotating disk with one flat surface in sliding contact with a hemispherically tipped rider or pin (3/16 in. rad.) under a normal load of 1 kilogram. The sliding velocity was 430 feet per minute.

COATING FORMULATION AND APPLICATION

The coating procedures reported in references 1 and 2 have been modified as a result of the studies reported herein. The major steps in the procedure are illustrated in figure 1, and the currently recommended practice is the following:

(1) Powder mixing. Powders of the ceramic binder material and CaF_2 are thoroughly mixed in the following proportion: one part of binder to three parts of CaF_2. (The powdered ceramic binder or frit has the composition 60 weight percent cobaltous oxide (CoO), 20 weight percent barium oxide (BaO), 20 weight percent boric oxide (B_2O_3) and is commercially available from the Ferro Corp., Cleveland, Ohio; however, small batches can be made in the laboratory according to the procedure described in the appendix.)

(2) Pebble mixing. Molybdic oxide (MoO_3) additions of 1 or 3 weight percent are made; then the mixed powders are wet-milled (1 cc of water/g of solids) in a pebble mill for 24 hours. The milled material is washed through a coarse sieve to remove the pebbles and then placed into a vacuum filter funnel to remove excess moisture. The resulting filter cake is a moist paste.

(3) Slurry preparation. The paste is thinned to a sprayable consistency by the addition of distilled water followed by thorough mixing in a high-speed blender. Experience has shown that water content is not critical, but about $1\frac{1}{2}$ cubic centimeters of water per gram of solids results in a slurry that is dilute enough to prevent clogging of spray equipment but not so dilute as to allow rapid separation of a water layer.

(4) Substrate surface preparation. Turned metal surfaces do not require surface roughening but finish ground or polished surfaces are wet-abraded with

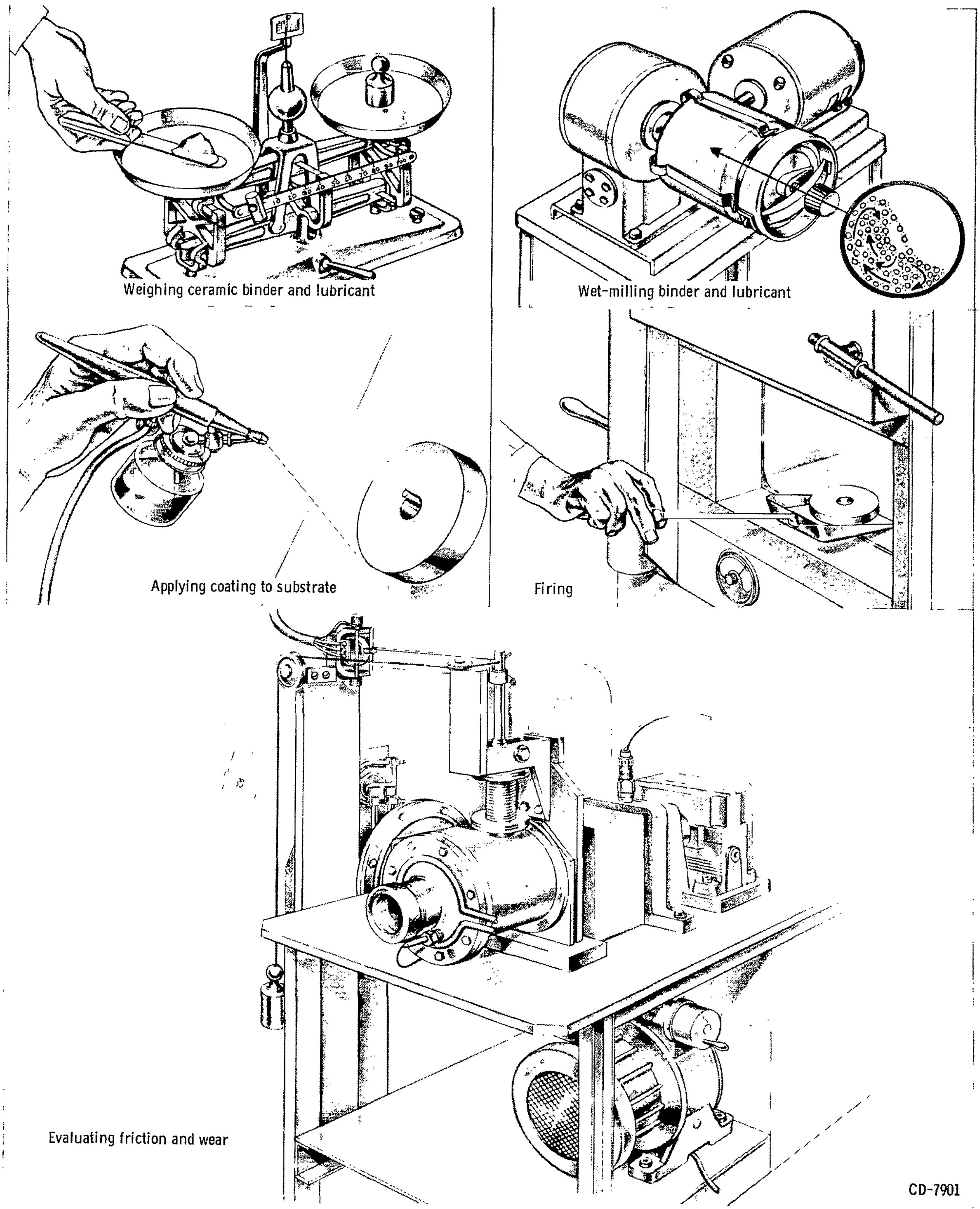


Figure 1. - Application and evaluation of ceramic-bonded calcium fluoride coating.

fine waterproof sandpaper to promote adherence of the spray coat. The metal specimens are heated in air at 1700° F until a thin, blue oxide film forms. (It is important not to heat too long. An oxide scale is very undesirable; only the thin, blue, transparent oxide film is desired. The blue color is caused by light interference and corresponds to an oxide film thickness of about 700 angstroms. Time at temperature varies with the mass of the specimen. For example, the solid friction disks ($2\frac{1}{2}$-in. diam. by $\frac{1}{2}$ in.) require 3 minutes at 1700° F; Timken rings ($1\frac{3}{8}$ in. o.d. and about $1\frac{1}{8}$ in. i.d.) require only 2 minutes at 1700° F.)

(5) Spraying. The lightly oxidized specimens are then sprayed with the lubricant slurry; an air brush is used to obtain a fine mist spray. The specimen surface temperature is held at about 150° F with an infrared lamp or a warm air blower to promote rapid evaporation of water from the spray coat. The coating is built up to a thickness of 0.001 to 0.002 inch by repeated passes of the spray.

(6) Firing. The as-sprayed coating is weakly bonded to the substrate. A strong fusion bond is developed by firing the coating at 2150° F until the ceramic binder becomes molten. The specimens are then allowed to cool. During cooling from 2150° to 1500° F, a cooling time of 1 to 2 minutes is satisfactory; the cooling rate below 1500° F does not appear to be critical. (The firing time is important; 3 minutes at 2150° F is satisfactory for $2\frac{1}{2}$-inch-diameter by 1/2-inch disks. Smaller parts would require a shorter firing time. Underfiring is indicated if the CaF_2 agglomerates. A properly fired coating is uniform, quite smooth, and is not powdery, but some CaF_2 powder should be freed when the surface is lightly scraped with an edged tool.)

(7) Surface finishing. The surface of the coating is generally satisfactory in the as-fired condition. Minor surface roughness can be removed by very light sanding with 6/0 grade garnet paper.

(8) Overlay application (optional). The overlay is applied by vigorously rubbing a finely powdered fluoride eutectic composition, which is 62 weight percent barium fluoride plus (BaF_2) 38 weight percent CaF_2, into the surface of the coating. This process reduces surface porosity without measurably increasing the coating thickness.

FRICTION AND WEAR APPARATUS AND EXPERIMENTAL PROCEDURE

The apparatus used in the friction and wear studies is shown in figure 2. A $2\frac{1}{2}$-inch-diameter rotating disk is placed in sliding contact with a hemispherically tipped rider (3/16 in. rad.) under a normal load of 1000 grams. The coatings are applied to the disk specimens only. The rider describes a 2-inch-diameter wear track on the disk. Sliding is unidirectional. The rider specimen is supported in the specimen chamber by a retaining arm that is gimbal bellows mounted to the chamber. A linkage at the end of the retaining arm away

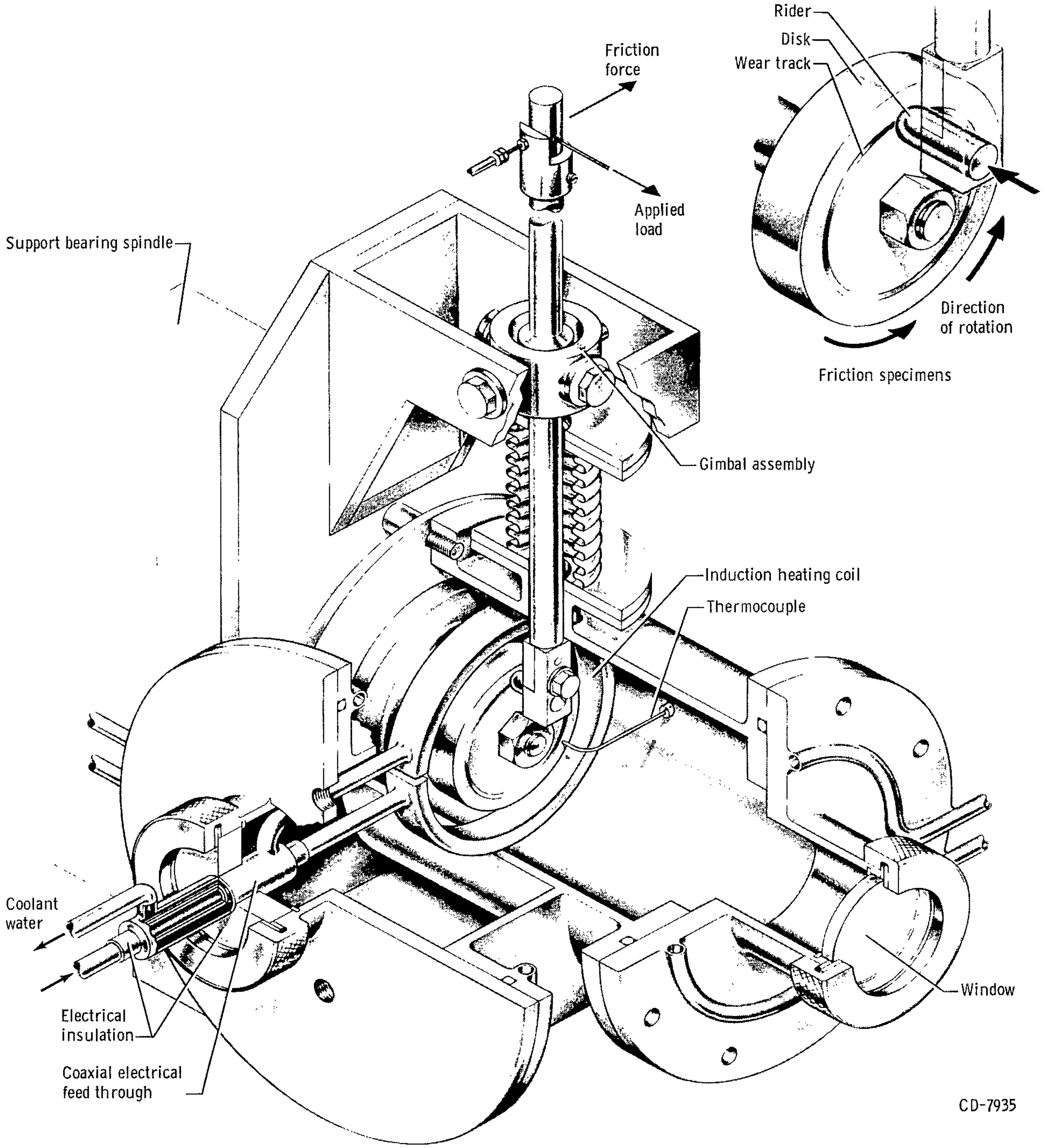


Figure 2. - High-temperature friction apparatus.

from the rider specimen is connected to a strain-gage assembly that is used to continually measure friction force during experiments. The load is transmitted through the same retainer arm in a direction normal to the friction force. Load is applied through a dead-weight loading system. In this program, the sliding velocity was maintained constant at 430 feet per minute. The duration of most experiments was 1 hour. Specimen materials in most cases were cast Inconel riders and Inconel X disks for experiments to 1500° F and René 41 riders and disks for higher temperatures.

RESULTS AND DISCUSSION

The effects of modifications in coating procedure and in coating composition on lubricating properties of ceramic-bonded CaF_2 were studied. The following modifications were evaluated:

(1) Pebble-milling

(2) MoO_3 additions

(3) Bentonite additions

(4) Eutectic fluoride overlay

(5) Surface polishing

(6) Preglazed wear tracks

Effect of Pebble-Milling

CaF_2 and the ceramic binder material were available in powder form; individual particles of powder were irregular in shape, but the particle size was typically 0.001 to 0.002 inch. Wet-grinding the CaF_2 and the binder in a pebble mill for 24 hours reduced the particle size to approximately 0.00005 inch and ensured the attainment of an extremely uniform mixture.

The effect of pebble-milling is given in table I. There was little effect on rider wear. The typical friction coefficients, however, were lower at 1000° and 1500° F for coatings prepared from milled powders.

Thorough mixing and a fine particle size are important because only the ceramic binder melts during firing; therefore, the distribution of CaF_2 in the as-fired coating is strongly influenced by the degree of uniformity of the coating in the as-sprayed but unfired condition. The friction coefficient is influenced by the ratio of CaF_2 to ceramic binder within the surface area of sliding contact. Accordingly a greater scatter in the values of the friction coefficient was observed with the more heterogeneous coatings prepared from unmilled powders.

TABLE I. - INFLUENCE OF PEBBLE-MILLING AND USE OF EUTECTIC FLUORIDE OVERLAY ON FRICTION AND WEAR PROPERTIES OF CERAMIC-BONDED CALCIUM FLUORIDE COATINGS

[Load, 1 kg; sliding velocity, 430 ft/min; coating thickness, 0.001 to 0.002 in.; basic coating composition, 73 percent CaF_2 + 24 percent binder + 3 percent MoO_3; binder composition, 60 percent CoO + 20 percent BaO + 20 percent B_2O_3; test duration, 1 hr.]

Temperature, °F	Coating modification	Rider wear rate, cu in./hr	Typical friction coefficient (a)	Comments
75	(b)	--------	0.40	Failed in 2 min
500	(c)	4.2×10^{-6}	0.40	------------------
	(d)	--------	.16	Coating had been run in at 1500° F
	(b)	2.6	.27	------------------
1000	(c)	4.1×10^{-6}	0.15	------------------
	(d)	1.8	.12	------------------
	(b)	.3	.10	------------------
1500	(c)	0.9×10^{-6}	0.20	------------------
	(d)	.9	.09	------------------
	(b)	.4	.13	------------------
1600	(c)	0.9×10^{-6}	0.20	------------------
	(d)	1.4	.20	------------------
	(b)	.9	.20	------------------

[a]In cases where run-in period with erratic friction was observed, tabulated friction coefficients represent typical values after friction coefficients stabilized.

[b]Coatings were prepared from milled powders and overlaid with thin burnished film of 62 percent BaF_2 + 38 percent CaF_2.

[c]Coatings were prepared from mixed but unmilled powders (0.001- to 0.002-in. average particle size).

[d]Coatings were prepared from milled powders (0.00005-in. average particle size).

Influence of Molybdic Oxide in Coating Composition

Small additions of MoO_3 often reduce the surface tension and increase the fluidity of molten ceramics (refs. 3 and 4). MoO_3 in the coating composition might improve wettability of the molten ceramic for both CaF_2 particles and the substrate metal during the firing operation. Therefore, small additions (1 and 3 percent) of MoO_3 were made to the basic coating composition just before pebble milling. The effect of these additions on the lubricating properties of the coating are given in table II (p. 8) and figure 3 (p. 9). All of the coatings in this group were prepared from powders that had been pebble-milled.

The addition of 1 percent MoO_3 to the basic coating composition had no large effect on wear up to 1700° F. The addition, however, was responsible for

TABLE II. - INFLUENCE OF MOLYBDIC OXIDE AND BENTONITE ADDITIONS ON FRICTION AND WEAR PROPERTIES OF CERAMIC-BONDED CALCIUM FLUORIDE COATINGS PREPARED FROM PEBBLE-MILLED POWDERS

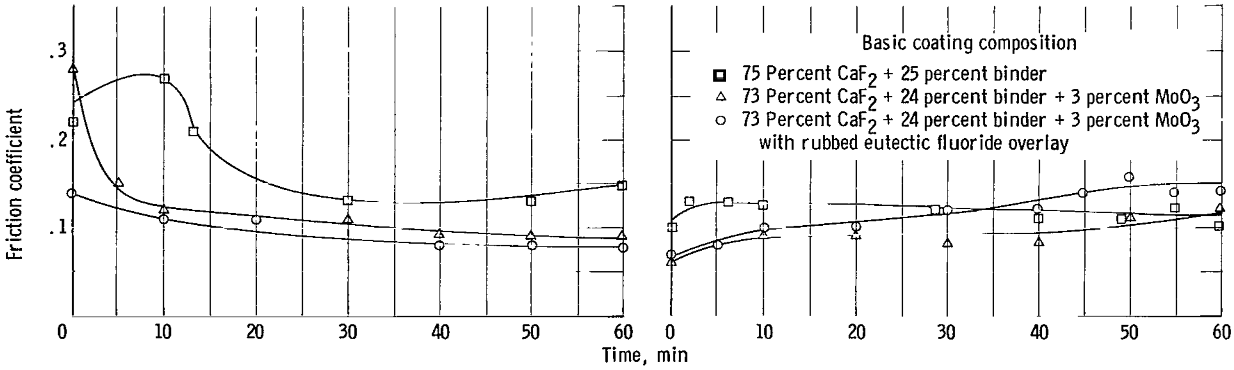
[Load, 1 kg; sliding velocity, 430 ft/min; coating thickness, 0.001 to 0.002 in.; basic coating composition, 75 percent CaF_2 + 25 percent binder; binder composition, 60 percent CoO + 20 percent BaO + 20 percent B_2O_3; test duration, 1 hr.]

Temperature, °R	Additions to basis coating composition, percent	Average rider wear rate, cu in./hr	Number of tests	Typical friction coefficient	Comments
75	3 MoO_3	--------	---	0.40	Failed in 2 min
500	None	3.0×10^{-6}	1	0.18 or 0.35	------------------
	1 MoO_3	1.2	1	.24 or .35	------------------
	3 MoO_3	--------	---	.16	Coating had been run in at 1500° F
1000	None	1.0×10^{-6}	2	0.15	------------------
	1 MoO_3	2.8	2	.10	------------------
	3 MoO_3	1.8	4	.12	------------------
	3 MoO_3 + 3 clay[a]	8.4	1	.15	------------------
1500	None	15.0×10^{-6}	1	0.12 or 0.10	------------------
	1 MoO_3	1.1	3	.10	------------------
	3 MoO_3	.9	3	.09	------------------
	3 MoO_3 +3 clay[a]	.9	1	.10	------------------
1600	None	2×10^{-6}	1	0.08	------------------
	1 MoO_3	.6	1	.15	------------------
	3 MoO_3	1.4	1	.20	------------------
1700	None	0.6×10^{-6}	1	0.15	------------------
	1 MoO_3	.7	1	.23	------------------
	3 MoO_3	--------	1	----	Failed in 5 min

[a]Bentonite suspending agent.

a decrease in the average friction coefficient at 1000° and 1500° F and an increase in friction coefficient at 1600° and 1700° F. At 1700° F the friction coefficient was 0.23 compared to 0.15 for the coating without MoO_3; however, rider wear was still very low.

Three-percent additions of MoO_3 to the basic coating composition resulted in lower friction coefficients at 1000° and 1500° F. Although the friction coefficient at 1600° F was 0.20 compared to 0.08 for the unmodified coating, rider wear was not increased. At 1700° F, coatings with 3 percent MoO_3 wore through almost immediately. The data of figure 3(a) show that, at 1000° F, a significant improvement in the run-in characteristics and a reduction in the average friction coefficient are obtained by the 3 percent MoO_3 addition. Corresponding data for 1500° F experiments are given in figure 3(b). At this



(a) At 1000° F. (b) At 1500° F.

Figure 3. - Influence of MoO_3 addition and use of rubbed eutectic fluoride (62 percent BaF_2 - 38 percent CaF_2) overlay on friction characteristics of ceramic-bonded CaF_2 coatings. Load, 1 kilogram; sliding velocity, 430 feet per minute; coating thickness, 0.001 to 0.002 inch; binder composition, 60 percent CoO + 20 percent BaO + 20 percent B_2O_3.

temperature, MoO_3 additions were not required to obtain stable friction characteristics.

The general effect of MoO_3 additions is to improve the coating at temperatures up to 1500° F, but the same additions lower the maximum temperature for effective lubrication. The maximum service temperature for the basic coating composition is 1900° F (ref. 2). The maximum service temperature after a 1 percent MoO_3 addition is 1700° F, after a 3 percent addition, it is 1600° F.

Effect of Bentonite Additions

Bentonite is a clay (largely composed of aluminum silicate) that is used as a suspending agent. It also improves adhesion between the as-sprayed coating and the substrate; the primary advantage is greater ease of handling of the as-sprayed parts. As indicated in table II, a 3 percent bentonite addition did not significantly influence the lubricating properties of the coating at 1500° F, but at 1000° F, rider wear was about four times higher than for the coating without bentonite.

Influence of Eutectic Fluoride Overlay

The combined effect of pebble-milling and a 3 percent MoO_3 addition was to reduce both the magnitude and the variability of the friction coefficient during the wear life of the coatings. At 1000° F, the problem of a high friction coefficient (~0.3) during the first few minutes remained. The problem was remedied by applying a rubbed film of a powdered fluoride eutectic composition (62 percent BaF_2 - 38 percent CaF_2) to the coating surface. Rider wear rate at 1000° F was reduced to about one-sixth the wear rate obtained without the rubbed overlay (table I). Figure 3(a) shows that the rubbed overlay reduces the initial friction coefficient at 1000° F from about 0.25 to 0.15 and reduces the average friction coefficient from about 0.15 to 0.10. Figure 3(b) shows that at 1500° F the overlay was not required to ensure a low initial friction

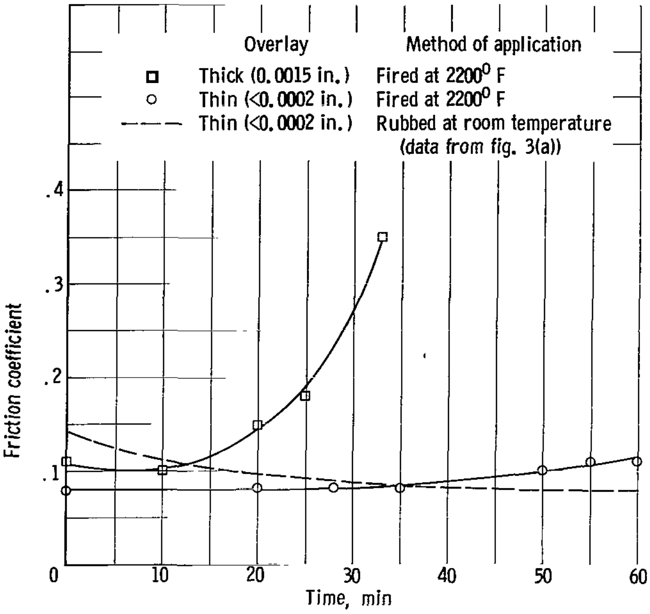


Figure 4. - Friction characteristics at 1000° F of ceramic-bonded CaF_2 coating with rubbed eutectic fluoride (62 percent BaF_2 - 38 percent CaF_2) overlay applied by various methods. Load, 1 kilogram; sliding velocity, 430 feet per minute; coating thickness, 0.001 to 0.002 inch; basic coating composition, 73 percent CaF_2 + 24 percent binder + 3 percent MoO_3.

coefficient with coatings that had been prepared from thoroughly milled powders.

Coatings with the overlay were also run at 75° and 500° F (see table I). A high friction coefficient (0.40) was observed at 75° F and the rider wore through the coating in 2 minutes (1740 cycles). At 500° F, rider wear was not prohibitive and the friction coefficient was 0.27. Even with the overlay, high surface temperatures are required to obtain low friction coefficients. At the sliding velocities used in this experiment (430 ft/min), the coating with 3 percent MoO_3 addition and an overlay could probably be used from about 500° to 1600° F.

The eutectic fluoride overlay was beneficial only when used as an extremely thin film. The effect of overlay thickness on the friction coefficient at 1000° F is shown in figure 4. A very thin film of less than 0.0002 inch applied either by spraying followed by firing or simply applied as a rubbed film was beneficial in obtaining a low initial friction coefficient and a stable friction coefficient during the experiments. A relatively thick overlay (0.0015 in.) that was applied by spraying then firing, caused a serious reduction in the wear life of the lubricant. Therefore, the convenient procedure of applying the eutectic fluoride as a rubbed film was the most efficient. The rubbed film was effective because the particles of fluoride powder were rubbed into the pores in the surface of the coating and in effect increased the ratio of solid lubricant to ceramic binder at the surface.

Influence of Polishing

In one attempt to reduce friction during run in, the coating surface was polished on a metallographic polishing wheel charged with a very fine grade of alumina. Microscopic examination showed that the polishing action removed the soft CaF_2 particles from the surface of the coating. During a 1 hour friction experiment at 1000° F, the friction coefficient during the first 30 minutes had a value characteristic of the ceramic binder or about 0.40 (fig. 5). It was not until wear exposed subsurface CaF_2 that the friction coefficient decreased to about 0.20.

Another attempt to modify the surface in the hope of reducing the initial friction coefficient was to fire polish the surface with an oxyacetylene flame.

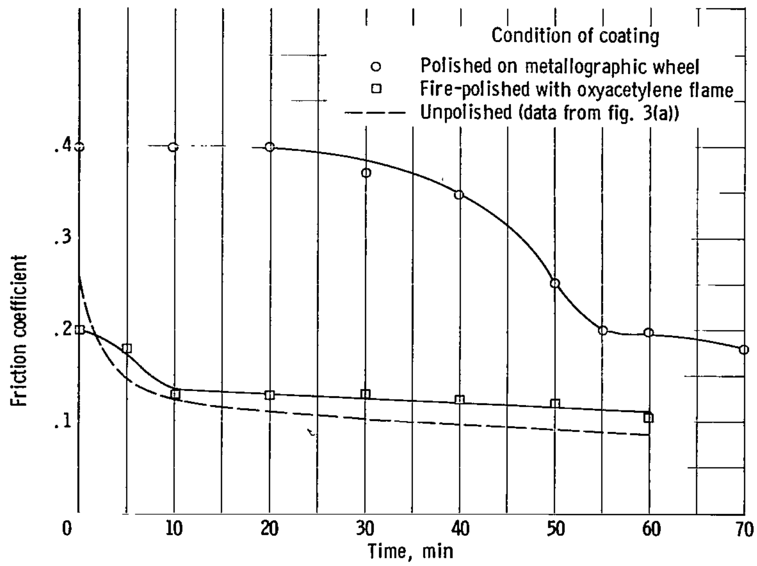


Figure 5. - Influence of flame-polishing and wheel-polishing on friction characteristics of ceramic-bonded CaF_2 coating at 1000° F. Load, 1 kilogram; sliding velocity, 430 feet per minute; coating thickness, 0.001 to 0.002 inch; basic coating composition, 73 percent CaF_2 + 24 percent binder + 3 percent MoO_3; binder composition, 60 percent CoO + 20 percent BaO + 20 percent B_2O_3.

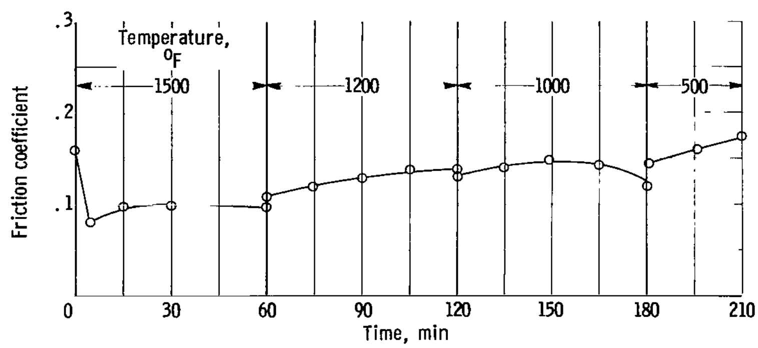


Figure 6. - Friction characteristics of ceramic-bonded CaF_2 coating at 1200°, 1000°, and 500° F when glazed wear track is first established at 1500° F. Load, 1 kilogram; sliding velocity, 430 feet per minute; coating thickness, 0.001 to 0.002 inch; basic coating composition, 73 percent CaF_2 + 24 percent binder + 3 percent MoO_3.

Figure 5 shows there was no significant improvement in the friction properties of the flame-polished coating.

Effect of Preglazed Wear Track

All of the coatings prepared from milled powders were consistently good at 1500° F. The wear tracks were very smooth and glazed after 1500° F experiments. Therefore it was of interest to determine whether the friction properties at lower temperatures would be improved by first establishing a glazed wear track at 1500° F. The results are shown in figure 6. A coating without an overlay was run for 1 hour at 1500° F, 1 hour at 1200° F, 1 hour at 1000° F, and finally for 10 minutes at 500° F. The friction coefficients at 1200° and 1000° F showed little variation, and extremely smooth sliding occurred. The friction coefficient at 500° F was 0.16. It is beneficial therefore to establish a burnished or glazed bearing surface at 1500° F prior to running at lower temperatures.

SUMMARY OF RESULTS

The present study of ceramic-bonded calcium fluoride (CaF_2) coatings was conducted in an attempt to determine the source of occasionally erratic friction characteristics and to determine what corrective measures were in order. A modified coating procedure, based upon the results of this program, is given in the body of this paper. The major findings of this study are the following:

1. In general, modifications in the coating composition or in the firing procedure that tend to refine or homogenize the distribution of CaF_2 throughout the binder stabilized the friction coefficient especially during the early or run-in stage.

2. The properties of the coatings were improved by the following steps in the coating preparation: (a) prolonged wet, pebble-milling of CaF_2-ceramic mix, (b) the use of a very finely atomized mist when spraying the coating on the substrate, and (c) careful control of the firing time and temperature.

3. The additions of (1 and 3 percent) molybdic oxide (MoO_3) to the coating composition were beneficial. These additions increased the fluidity of the molten ceramic binder during firing and appeared to enhance the wettability of CaF_2 particles by the molten ceramic.

4. At temperatures from 1000° to 1600° F, further benefit was derived by the application of a very thin rubbed film of a binary fluoride eutectic composition (62 percent BaF_2 - 38 percent CaF_2) to the surface of the ceramic-bonded coating. The beneficial effect was smoother sliding and a lower initial friction coefficient.

5. Much better lubrication was obtained at 1000° F and lower temperatures if a well-glazed wear track was first established by running the specimens at 1500° F.

6. The addition of 3 percent bentonite clay (a suspending agent) to the composition improved the spraying characteristics, had no influence on the friction characteristics of the fired coating, but appeared to increase rider wear, especially at 1000° F.

7. Fire-polishing the bonded coating with an oxyacetylene flame had no effect on the lubricating properties of the coatings.

8. Finishing the bonded coating on a polishing wheel charged with Al_2O_3 polishing compound was detrimental. The polishing action apparently removed

the softer CaF_2 particles and left only ceramic binder material at the surface. Friction coefficients of the polished coatings were high and characteristic of the binder until the surface layer eventually wore away exposing more CaF_2.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, December 16, 1964.

APPENDIX - PREPARATION OF THE CERAMIC BINDER USED TO BOND CaF_2 TO NICKEL-BASE SUPERALLOYS

The raw materials for the ceramic binder are reagent grades of cobalt oxide (Co_3O_4), boric acid (H_3BO_3), and barium hydroxide octahydrate (Ba(OH) ($Ba(OH)_2 \cdot 8H_2O$). These compounds will decompose to CoO, B_2O_3, and BaO, respectively, at the temperatures indicated in the following assumed overall decomposition reactions:

$$Co_3O_4 \xrightarrow{1600^\circ\ F} 3CoO + \frac{1}{2} O_2$$

$$2H_3BO_3 \xrightarrow{365^\circ\ F} B_2O_3 + 3H_2O$$

$$Ba(OH)_2 \cdot 8H_2O \xrightarrow{1020^\circ\ F} BaO + 9H_2O$$

The following procedure yielded the best results:

(1) A mixture of Co_3O_4, H_3BO_3, and $Ba(OH)_2 \cdot 8H_2O$ is prepared with the following weight percentages: 45.6 percent Co_3O_4, 25.3 percent H_3BO_3, and 29.1 percent $Ba(OH)_2 \cdot 8H_2O$.

(2) The mixture is melted in a porcelain crucible at 2200° F until the reactions cease and a uniform quiescent melt is obtained. The calculated composition after completion of the decomposition reactions is 60 percent CoO, 20 percent B_2O_3, and 20 percent BaO.

(3) The melt is poured slowly into cold water to form friable shotlike globules, which are then filtered, dried, and ground to pass through a 200-mesh screen. An alternative method is roll-quenching followed by grinding. (Roll-quenching consists of pouring the melt on water-cooled stainless-steel rolls.)

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